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Final Report

SECONDARY ION MASS SPECTROMETRY CHARACTERIZATION OF IMPLANTS INTO GAAS AND GAP FOR WAVEGUIDES AND INTEGRATED OPTICS APPLICATIONS

Prepared by:

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using a Pearson IV computer fitting routine.

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A significant result of this effort was the establishment of secondary ion mass spectrometry (SIMS) technologies for these materials. The use of our implants of known isotope and fluence provided calibration standards for the quantification of SIMS. We established SIMS relative sensitivity factors for both oxygen and cesium primary ion beams and positive and negative secondary ion mass spectrometry, using the variety of ions implanted. Another significant result of this research program was the measurement of range-energy and range straggle-energy tables and curves for the ions and materials studied. We used special SIMS bulk analysis techniques to measure the impurities in the materials that we used on the program, and found impurities other than those quoted as the dopants (Si, S, SN, Zn, etc.).



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EXECUTIVE SUMMARY

We implanted H, He, and most dopant elements into GaAs, GaAsP, GaP, GaSb, InP, InAs, InSb, and LiNbO3 at energies from 10 to 700 keV, and a few elements up to 2.4 MeV, in random and channeling orientations, annealed some of these implants, and measured their depth distributions using secondary ion mass spectrometry (SIMS). We calculated values of $R_{\rm m}$, $R_{\rm p}$, $\Delta R_{\rm p}$, γ_1 , β_2 , using an LSS formulation, and using TRIM (for a few), a Monte Carlo calculation, and measured these same profile parameters for the experimental profiles using a Pearson IV computer fitting routine.

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A number of collaborators contributed to the work summarized here, via various publications and measurements. We especially acknowledge D. Jamba, J. Zavada, S. Pearton, S. Novak and the SIMS staff at CHARLES EVANS & ASSOCIATES, D. Ingram, P. Pronko, P. Thompson, R. Laidig and J. Comas

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I. INTRODUCTION AND STATEMENT OF PROBLEM STUDIED

The objectives of this program were to provide understanding for the Army and DoD of III-V semiconductor materials and devices that can be fabricated in these materials as a result of the energy deposition or momentum transfer of energetic light ions via alteration of the properties of the materials. Affected properties include changes of the index of refraction, diffusion coefficients, or electrical characteristics (e.g., by defects, vacancies, scattering centers, etc.), as well as direct effects of changes in the electrical (semiconducting) properties of the crystalline material caused by the presence of the implanted atoms as n- or p-type dopants (donors or acceptors). In addition, information was needed about changes of all of the above effects caused by thermal processing of either short (seconds) or long (minutes) duration. These III-V materials were single crystals of one material or combined material structures (heterostructures), for example, superlattices.

The pursuit of these general objectives entails the implantation of elements as ions from throughout the periodic table, but because of the focus on III-V materials and waveguide applications, we were primarily concerned with the light ions ^{1}H , ^{2}H , and ^{4}He , and those from columns II, IV, and VI of the periodic table, especially Be, Mg, Ca, Zn, Sr, Cd, C, Si, Ge, Sn, O, S, Se, and Te. The ion energy range of interest was large because of the varied applications, including wave guiding structures in the infrared (tens of μm) and deep and shallow device structures (0.1 to

 $1.0 \, \mu m)$.

To meet these goals, we first obtained polished wafers of four III-V materials, GaP, GaAs, InP, and InSb, from a variety of sources. These materials were obtained both undoped and highly n-doped (> 10^{18} cm⁻³), the latter for waveguide fabrication via carrier removal. Crystals having the commonly available orientations (100) and (111), as well as (110) when possible, were obtained for channeling studies, an alternative method to MeV implantation to achieve deep penetration. During the course of this program, we received samples of InAs and GaSb from S. Pearton of ATT Bell Labs, for which we are grateful. We implanted the ions listed above at energies between 50 and 2000 keV in random and channeling orientations (the latter by aligning the desired crystal direction using Rutherford backscattering), at fluences between 5×10^{12} and 10^{14} cm⁻², or higher fluences for the light ions ¹H, ²H, and ⁴He, as appropriate for waveguide fabrication by carrier removal, namely, 10¹⁵ to 10¹⁶ cm⁻². We then annealed selected samples using furnace or rapid lamp annealing at temperatures appropriate for each material. The melting temperatures for GaSb and InSb are so low that little annealing effect could be achieved. GaP can withstand the highest temperatures. However, annealing temperatures were limited to 800°C for all materials using furnace annealing, and to 900°C using rapid lamp annealing.

II. SUMMARY OF THE MOST IMPORTANT RESULTS

We implanted H, He, and most dopant elements into GaAs, GaAsP, GaP, GaSb, InP, InAs, InSb, and LiNbO₃ at energies from 10 to 700 keV, and a few elements up to 2.4 MeV, in random and channeling orientations, annealed some of these implants, and measured their depth distributions using secondary ion mass spectrometry (SIMS). We calculated values of R_m , R_p , ΔR_p , γ_1 , β_2 , using an LSS formulation, and using TRIM (for a few), a Monte Carlo calculation, and measured these same profile parameters for the experimental profiles using a Pearson IV computer fitting routine.

This work included studies of: characterization of Si implantation into GaAs in random and channeling orientations at energies from 10 keV to 6.0 MeV; a general description of as-implanted and annealed depth profiles of dopant implants (especially Be and Si) into GaP, GaAs, InP, and InSb; illustrations of the effects of heterostructures, including superlattices, on the redistribution of mobile impurities (H and Be) during thermal processing; brief illustrations of implants in other III-V technologies (GaAs/Si and GaAsP); lanthanide rare earth elements in III-V crystals; as-implanted and annealed MeV implants in III-V materials; characterization of proton exchanged LiNbO3; and redistribution of implanted H in LiNbO3.

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III. LIST OF ALL PUBLICATIONS

<u>Publications</u>: Publications and conference presentations that resulted from this program are listed below.

- 1. J.M. Zavada, H.A. Jenkinson, R.G. Sarkis, and R.G. Wilson, "Hydrogen depth profiles and optical characterization of annealed, proton -implanted n-type GaAs." J. Appl. Phys. <u>58</u>, 3731-3734 (1985)
- 2. R.G. Wilson, "Implant profiles in GaP, GaAs, InP, and InSb: influence of furnace and rapid thermal annealing," Proc. SPIE FLA 1987
- 3. P.E. Thompson, R.G. Wilson, D.C. Ingram, and P.P. Pronko, "Atomic profiles of high energy (MeV) Si implanted into GaAs," Mat. Res. Soc. Proc. 93, 73-77 (1987)
- 4. J.M. Zavada, R.G. Wilson, and S.W. Novak, "Accumulation of implanted hydrogen at the superlattice/substrate interface of a GaAs-AlAs multilayer structure," EESDERC Bologna, Italy, Sept, 1987
- 5. J.M. Zavada, R.G. Wilson, S.W. Novak, A.R. von Neida, and S.T. Pearton, "Redistribution of implanted hydrogen in p* GaAs(Zn) and n* GaAs(Si) single crystals," Proc. MRS Boston, Dec 1987
- 6. S.W. Novak and R.G. Wilson, "SIMS measurements of impurities and dopants in AlGaAs heterostructures," Proceedings of the Sixth International Conference on Secondary Ion Mass Spectrometry (SIMS VI), Versailles, France, September 1987, pp. 303-06
- 7. R.G. Wilson, S.W. Novak, and J.M. Zavada, "Redistribution of implanted hydrogen and substrate dopants in annealed (100 to 600 °C) substrates of GaP(S), InP(S), InP(Sn), GaAs(Si), and GaAs(Zn), "Inst. Phys. Conf. Ser. 91, 475-77 (1987)
- 8. R.G. Wilson, S.W. Novak, and J.M. Zavada, "Depth distributions of Be and Si implanted into GaP, InP, and InSb, after implantation and after furnace or flash lamp annealing, compared with GaAs," Inst. Phys. Conf. Ser. 91, 479-83 (1987)
- 9. R.G. Wilson, J.M. Zavada, S.W. Novak, and S.P. Smith, "Depth profiles and redistribution during annealing of 300-keV hydrogen (protons) implanted into an AlAs/GaAs superlattice," Inst. Phys. Conf. Ser. 91, 485-88 (1987)
- 10. S.W. Novak and R.G. Wilson, "Two- and three-dimensional characterization of AlGaAs heterostructures using SIMS," Inst. Phys. Conf. Ser. 91, 597-600 (1987)
- 11. R.G. Wilson, D.M. Jamba, D.C. Ingram, P.P. Pronko, P.E. Thompson, and S.W. Novak, "Ranges, straggles, and shape factors of 20-keV through 6 MeV random and channeled Si implants into GaAs, unannealed and annealed," Inst.Phys, Conf. Ser. 91, 809-12 (1987)
- 12. J.M. Zavada, R.G. Wilson, and J.Comas, "Redistribution of H and Be in GaAs/AlAs multilayer structures with post implantation annealing," J.Appl. Phys. 65, 1968-71 (1989)
- 13. R.G. Wilson and D.B. Rensch, "Implantation and characterization of III-V materials," (Invited) Annual Materials Research Society Meeting, Sept., 1987, Boston, MA, J. Materials Research Society Vol., page (1989)
- 14. J.M. Zavada, R.G. Wilson, and J. Comas, "Ion beam processing of multilayer semiconductor structures, SPIE Symp. on Laser Technology in Industry, Porto, Portugal, 6-8 June 1988
- 15. J.M. Zavada, S.J. Pearton, R.G. Wilson, C.S. Wu, M. Stavola, F. Ren, J. Lopata, W.C. Dautremont-Smith, and S.W. Novak, "Electrical effects of atomic hydrogen incorporation in GaAs-on-Si," J. Appl. Phys. 65, 347-53 (1989)

16. R.G. Wilson, "Implantation range statistics in III-V materials," (Invited) ECS Conference on Ion Implantation in Compound Semiconductors, Hollywood, FL, 16-20 Oct 1989. To be published in the ECS Conference Proceedings.

17. R.G. Wilson, J.M. Zavada, and S.W. Novak, "As-implanted and annealing behavior of 0.20, 0.35, and 1.0 MeV ¹H and ²H implants into InP, and comparison with GaAs," (Invited) Workshop on Hydrogen Effects in InP and Related Compunds, Lannion, France, 24-25 October 1989

18. R.G. Wilson, S.W. Novak, J.M. Zavada, A. Loni, and R.M. De La Rue, "SIMS depth profiling of proton-exchanged LiNbO3," J. Appl. Phys. 15 Dec, xxx (1989)

19. R.G. Wilson, S.W. Novak, J.M. Zavada, A. Loni, and R.M. De La Rue, "SIMS of H:LiNbO3," Secondary Ion Mass-Spectrometry SIMS VII, H. ,Eds. [Wiley, NY, 1990] pp. xxx-xxx (Monterey,

CA, Sept 1989) (to be published soon)

20. A manuscript has been prepared for submission to J. Appl. Phys., titled "Systematics of SIMS relative sensitivity factors versus electron affinity and ionization potential for a variety of matrices determined from implanted calibration standards for about fifty elements," by R.G. Wilson and S.W. Novak, that acknowledges ARO support for the work in III-V materials.

IV. LIST OF ALL PARTICIPATING SCIENTIFIC PERSONNEL

DEGREES AWARDED DURING THIS REPORTING PERIOD:

None

SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT:

CHARLES EVANS & ASSOCIATES

Scott M. Baumann Richard J. Blattner Dr. Roger J. Bleiler Dr. Paul K. Chu Daniel Cudworth Ramon DeSantiago Dr. Charles A. Evans, Jr. Charles J. Hitzman Craig G. Hopkins Igor Ivanov Dr. Clive M. Jones Donald Martin Dr. Charles Kirschbaum Dr. Gavle Lux Dr. Steven W. Novak James C. Norberg Dr. Steven S. Smith Elizabeth M. Strathman Shaun D. Wilson

HUGHES RESEARCH LABORATORIES

Dr. Robert G. Wilson

V. RESEARCH EFFORT

Communication:

Communication between R. Wilson in California and J. Zavada in London regarding this contract program was carried out via many phone calls, FAX transmissions, and data packages mailed via the New York APO address during 1986, 1987, 1988, and 1989.

Wilson met with M. Littlejohn at the International Symposium on GaAs and Related Compounds in Hraklion. Crete, during September, 1987, where they discussed results and plans for future work. Wilson met with Littlejohn in Malibu, CA, during March of 1988 to discuss progress and future plans for work on this contract.

Wilson and Zavada met in Mailbu, CA, during December of 1987 and December of 1988 to discuss detailed technical work, to review progress, and to plan future work on this contract effort. Wilson met with Zavada once in London in Apr 1987, and once in France at the H in InP Workshop in Oct 1989 to discuss in detail technical work for this contract.

Wilson visited Charles Evans and Associates in Redwood City, CA almost once each month during the more than three years duration of the program, where he and staff members of Evans and Associates discussed work to be done and work completed, and worked together to carry out many SIMS analyses on samples prepared for the contract. S.W. Novak was the primary Evans and Associates staff member involved in this interaction and contributed greatly to the work and results of this effort.

Contract Business: A no cost time extension was requested and granted for this program. The modified contract ending date was 30 Oct 1989, with the final report due 60 days after that. This modified date included the time of the ERO/ARO sponsored H:InP workshop in Lannion, France, at which an invited paper was presented on work performed on this contract.

Samples Sent to Other Researchers for Measurements, and Type of Meaurement

The following implants were performed and mailed to H. Jenkinson at Picatinny Arsenal for carrier removal/optical measurements:

```
He 152 250 keV 1.0E14 cm<sup>-2</sup> into InP(Sn) He 153 250 keV 1.0E14 cm<sup>-2</sup> into InP(S) He 154 250 keV 1.0E14 cm<sup>-2</sup> into InP(Zn) D 227 300 keV 5.0E15 cm<sup>-2</sup> into GaAs(Zn) + 100, 200, 300, 400, 500, 600°C Anneals D 230 100 keV 5.0E15 cm<sup>-2</sup> into GaAs/Si epi H 3590 100 keV 1.0E16 cm<sup>-2</sup> into LiNbO<sub>3</sub> D 282 100 leV 1.0E16 cm<sup>-2</sup> into LiNbO<sub>3</sub>
```

Samples sent to other workers:

2 Be, 3 Zn, 4 Si, & Se asimplanted and annealed most 200 keV, 1.0E14 cm⁻² to Prof. Henry Lozykowski
Dept. Electr. & Computer Engr.
Ohio Univ. Athens, OH 45701
(614) 593-1587

optical electr.

Special Effort - Impurity Analysis in Spectrum Technology GaAs

An impurity analysis was performed using SIMS on four pieces of bulk LEC GaAs grown by Spectrum Technology. This effort was requested by M. Littlejohn, for ARO. Pieces from seed and tang of two boules were studied (ST 045, 053, 121, and 157). The impurities analyzed were B, C, O, Na, Mg, Si, S, Cr, Mn, Fe, Cu, Zn, Se, In, and Te. Several other pieces of LEC GaAs and one piece of HB GaAs were analyzed at the same time. The results for the ST material were comparable with other material and no impurity was unusually high. B was higher than for HB material but comparable with other LEC material that was also grown in a pBN crucible.

Si Implanted into GaAs

We implanted Si into GaAs at energies from 10 keV to 6.0 MeV in random and <110> channeling orientations, annealed some of these implants with proximity caps at 750 or 800°C for 15 or 20 min in a furnace, or for 10 or 15 s in a flash lamp annealer, and measured atom depth distributions using SIMS and some electrical profiles using C-V [3,8,11]. Conclusions from this work include: Si atoms do not redistribute during furnace anneals up to 800°C or during flash lamp anneals up to 1000°C, for fluences from 10¹² to 10¹⁴ cm⁻²; maximum Si density in <110> channeled regions is 2x10¹⁶ cm⁻³; <110> channeled depths are about 1 μ m at 20 or 40 keV and up to 12 μ m at 6 MeV; SIMS detection sensitivity for ³⁰Si in GaAs is about 1x10¹⁴ cm⁻³ (3xBG); experimental ranges (R_p) are 0 to 20% deeper than LSS calculations, and experimental range straggles ($\Delta R_{\rm p}$) are about 50% greater than LSS calculations at lower energies, but approach the same values as energy increases, until they converge at a few MeV; <110> channeled profile shape varies with ion energy, having three peaks at 6 MeV and relatively constant Si density from 3 to 11 μ m. Random and channeled MeV Si implants are illustrated in Figs. 2 and 3. Random and channeled range and range straggles are illustrated vs energy in Fig. 4; note the changes in slope at about 1 MeV. R_c is the depth of the channeled peak in the profile and R_{max} is the maximum range of the channeled ions. Figure 1 shows how Be or g can be used to compensate the deep tail on Si implants to sharpen the n-type profile and improve the electrical characteristics of FETs -- taken from publication 13, but this device portion of the work was not funded by ARO.

Implants into III-V Materials

We implanted the elements Be, Mg, Zn, Cd, C, Si, Ge, Sn, O, S, Se, Te, 1 H, and 2 H into GaS, GaP, and InP, and some of them into GaSb, InSb, and InAs, and annealed the more commonly used combinations with proximity caps in a furnace or flash lamp annealer [7,8]. Most elements were also channeled into the <110> direction of GaAs and InP, and the <111> or <100> direction of GaP. We measured their depth distributions using 0_2 or Cs

SIMS, and in some cases, using C-V. We calculated R_m , R_p (μ), ΔR_p (σ), γ_1 , and β_2 using an LSS formulation, and determined the corresponding experimental values using a Pearson IV computer fitting routine. The agreement is only fair because the LSS formulation assumes amorphous targets and the experimental data are for crystalline material; the agreement becomes much better when crystalline targets are pre-amorphized by self implants.

For SIMS, we measured the relative sensitivity factors (RSF) for quantification, the detection limits or sensitivities, the profile dynamic range, and molecular ion intensity comparisons. Various illustrations of the results are shown in Figs. 5 through 17. Conclusions from this work include: for normal implant conditions (channel or source/drain and 800°C), Be redistributes most in InP, less in GaAs, and little or not at all in GaP; Mg is similar to Be; Si does not redistribute in any III-V material, Ge is similar to Si, for fewer cases studied; and Sn diffuses in GaAs and possibly in other materials. The depth profile of associated damage in these implants is an issue, but is not dealt with here because of the difficulty of experimental measurement. Redistribution of implanted H into III-V materials is illustrated in Fig. 18 for GaAs. The H migrates deeper into the bulk at a density of about $3x10^{18}$ cm⁻³ with a strong temperature dependence, much as for thermal diffusion, but probably also depending on the diffusion and annhilation of defects. A comparison of the effects of furnace and flash lamp annealing is illustrated in Fig. 19 for GaP. Similar results were observed for GaAs and InP.

Heterostructures and Interfaces

We implanted several heterostructures and supperlattices and profiled both the matrix and impurity using SIMS [9,10]. An example is shown in Figs. 20 and 21, where an AlAs/GaAs structure implanted with H is shown via the Al depth profile (Fig. 20), which is disordered and collapses for a 500°C thermal treatment. The implanted H redistributes to the interface between the superlattice and the GaAs substrate as shown in Fig. 21, where it still remains after a 700°C thermal treatment. Similar superlattices are shown in Figs. 22 and 23, but grown on a GaAs buffer layer, and implanted with both H and Be. Here, both the H and Be redistribute to the buffer/substrate interface. However, the Be redistributes less and only significantly for a temperature of 600°C.

Other III-V Materials Technologies

We implanted Si-doped GaAs on Si (MBE) epi wafers with H and profiled the Si and H as a function of annealing temperature to 700° C [15]. The Si profile is unchanged and the H redistributes to the GaAs/Si interface where it remains greater than 10^{20} cm⁻³ even for the 700° C anneal, indicating that the strain (or defects at the interface) remains after this anneal temperature. These results are illustrated in Fig. 24. We implanted GaAsP with a variety of dopants and other elements and profiled them using SIMS. The results are similar to those for GaP and GaAs.

Rare Earth Implants into III-V Materials

We implanted many of the rare earth elements (Sc, Y, La, Ce, Nd, Sm, Eu, Tb, Dy, Ho, Er, Tm, Yb) into GaAs and InP in random and <110> channeling orientations and profiled them using SIMS. There is some interest in doping InP-based materials with these materials, esp. Er. The results confirm that these elements can be implanted in our standard implanter and that the depth profiles are consistent with those of surrounding elements. Random ranges are 70 to 90 μ m for 300 keV. <110> channeled ranges are 2 to 3 μ m for 300 keV. Maximum <110> channeled densities are about 3×10^{15} cm⁻³. Examples of random and channeled profiles are shown in Figs. 25 and 26, respectively, for Sm in GaAs.

H in III-V Materials

²H shows the redistribution of H in III-V materials better than ¹H because of the 100x better detection capability of SIMS for ²H.

III-V materials doped with H using plasmas or ion implantation plus anneals result in the same depth distributions.

H concentration is $\sim 100x$ higher in p-type (Zn-doped) III-Vs than in n-type (S-, Si-, or Sn-doped) III-Vs.

LiNb0₃

LiNbO $_3$ is another material that is important for wave guides. Work on LiNbO $_3$ was emphasized during the latter part of this contract, including samples of hydrogen-exchanged LiNbO $_3$ received from the University of Glasgow. A special SIMS technology was developed to profile this dielectric (charging) material. We also annealed a few representative implants in LiNbO $_3$ at 600° C/10min to determine the temperature stability of implants in LiNbO $_3$ under this one condition. The implants selected were H, B, F, P, Cl, Ge, Sb, and Te. Only H showed any redistribution upon annealing at 600° C, and the H redistibuted enough that no measurable H depth profile remained. LiNbO $_3$ was SIMS profiled at ~ 1.0 nm/s $\rightarrow 10$ µm/hr $\rightarrow \sim 1$ K\$/profile

MeV Implants

The MeV implant phase of this program consisted of implanting high energy H, He, Be, Si, and Ge into various materials, annealing them at various temperatures up to 750°C, and measuring the as-implanted and annealed depth distributions using SIMS.

The following MeV implants were performed:

plus 1.0, 2.0, 4.0, and 6.0 MeV Si into GaAs in random and <110> channeling orientations, in collaboration with Universal Energy Systems, and measured also for C-V electrical profiles by Naval Research Laboratory into materials that included GaAs, GaAs(Si), GaP(S), InP(S), InP(Fe), InSb, GaSb(few), and LiNbO₃. The as-implanted and annealed (750°C/20 min) Be profiles were measured. The as-implanted and annealed (300°C) He profiles were measured. The as-implanted and some annealed (300 and 500°C,) H samples have been profiled. Additional anneal temperatures would be useful if the program is renewed. The 1-MeV H depths are all of the order of 15 μ m. The ranges of 0.7 MeV Be and 2.0 MeV Si are about 1.6 μ m. Representative depth profiles are illustrated in Figs. 27 and 28. annealed profiles are different in the various materials. For example, the H has redistributed very little in the GaP compared with InP and GaAs, which are different from one another as well. The results for annealed Be are similar, namely, Be has not redistributed in GaP, has redistributed in both directions to form a rounded hump in InP, and has formed a non symmetric and structured profile in GaAs.

Other Efforts

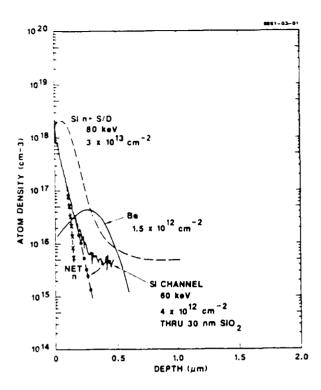
Work on GaP was emphasized during the latter part of the contract.

Implants of the lanthanide rare earths were also successfully carried out, into GaAs and InP.

Special work was performed on H and D plasma diffused samples of GaAs received from Bell Laboratories.

Samples of GaAsP provided through S.W. Novak of Charles Evans and Associates were implanted, annealed, and profiled using SIMS.

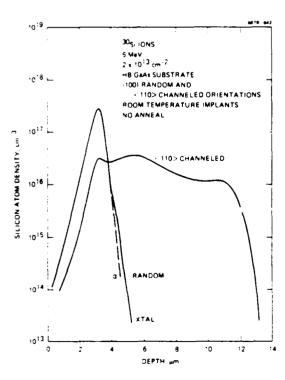
Samples of InAs and GaSb received from Bell Laboratories were implanted with a variety of elements and profiled using SIMS.

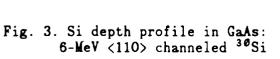


CONCENTATION (atous/cc)

Fig. 1. Be implant compensation of Si channel tail

Fig. 2. Si depth profiles in GaAs: 4-MeV random ^{28,29,30}Si





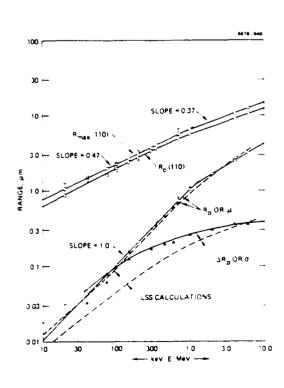
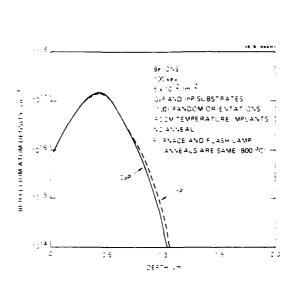


Fig. 4. Range and range straggle for Si in GaAs



Be CNS
120 keV
1, 1014 junil
34P Be CNS
120 keV
1, 1014 junil
34P AND INP SUBSTRATES
1301 RANDOM ORIENTATION
RICOM TEMPERATURE IMPLANTS
NO ANNEAL

1015
1015
1016
24P
25PTH Jm

Fig. 5. Low fluence profiles of Be in GaP and InP

Fig. 6. Moderate fluence profiles of Be in GaP and InP

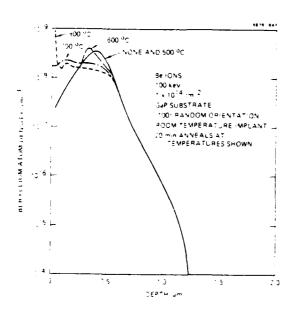


Fig. 7. Annealed profiles of Be in GaP

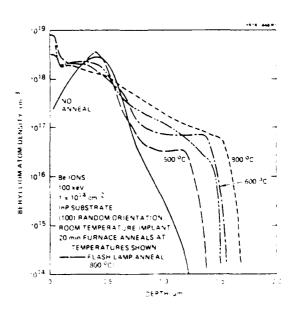


Fig. 8. Annealed profiles of Be in InP

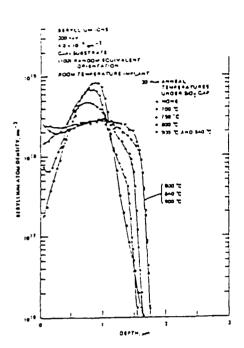


Fig. 9. Annealed profiles of Be in GaAs

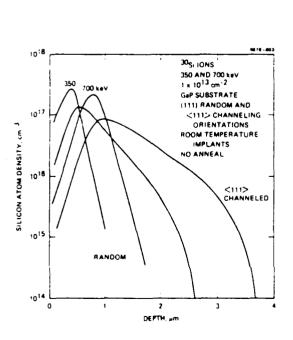


Fig. 11. Channeled profiles of Si in GaP

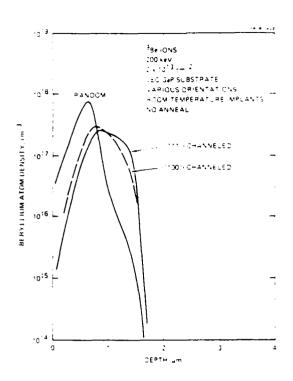


Fig. 10. Channeled profiles of Be in GaP

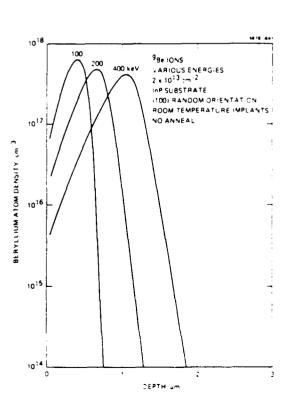


Fig. 12. Random profiles of Be in InP

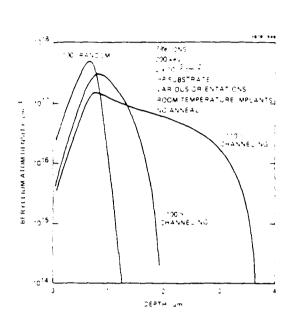


Fig. 13. Channeled profiles of Be in InP

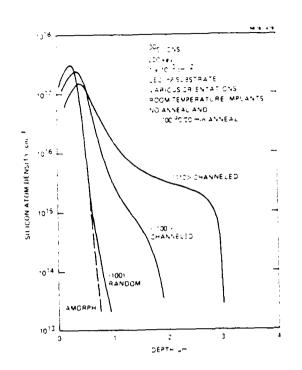


Fig. 14. Channeled profiles of Si in InP

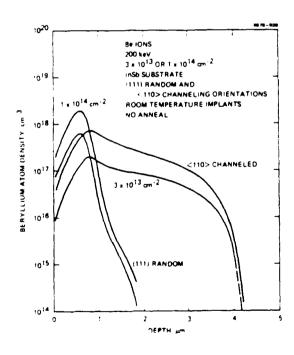


Fig. 15. Channeled profiles of Be in InSb

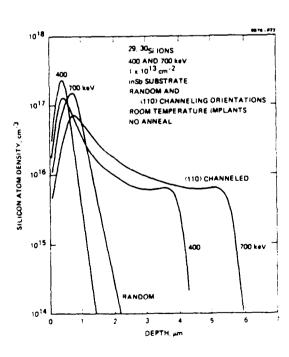
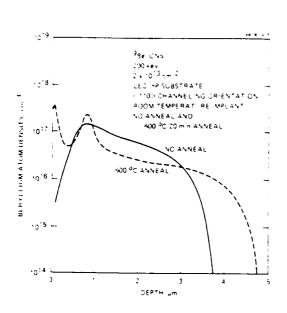
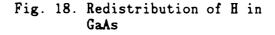


Fig. 16. Channeled profiles of Si in InP



8878 - 108 .021 'H IONS 300 keV 1 x 10¹⁶ cm⁻² 1020 GaAs (Zn) SUBSTRATE (100) RANDOM ORIENTATION HYDROGEN ATOM DERSITY, \cos^{-3} ROOM TEMPERATURE IMPLANT 20 min ANNEALS AT TEMPERATURES SHOWN IN OC 1019 1018 1017 300 300/60 min 600 1016 0 10 20 30 40 DEPTH, µm

Fig. 17. Annealed channeled profile of Be in InP



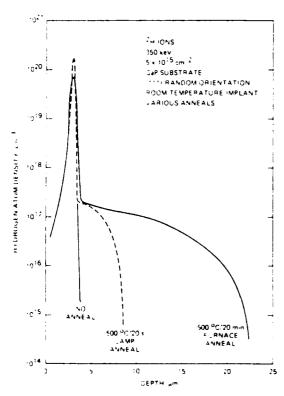


Fig. 19. Furnace vs flash lamp annealing for H in GaP

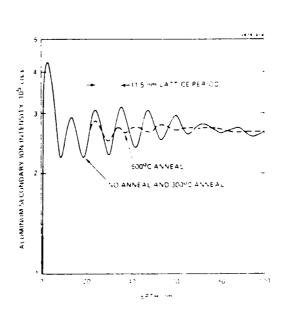


Fig. 20. Layers in an AlAs/GaAs superlattice

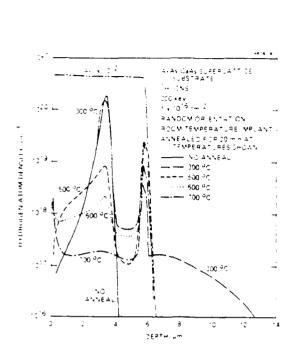


Fig. 21. H redistribution in an AlAs/GaAs superlattice

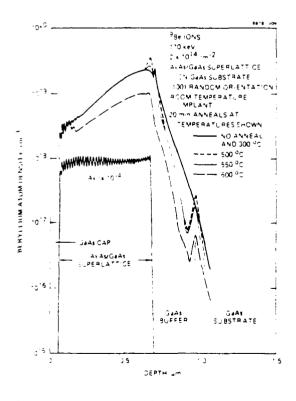


Fig. 23. Redistribution of Be in a superlattice/buffer

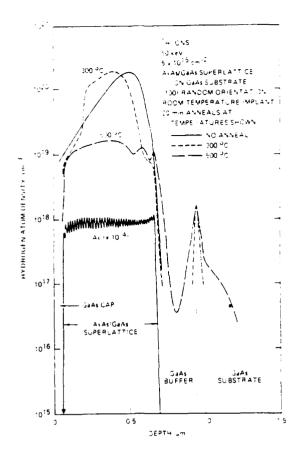


Fig. 22. Redistribution of H in a superlattice/buffer

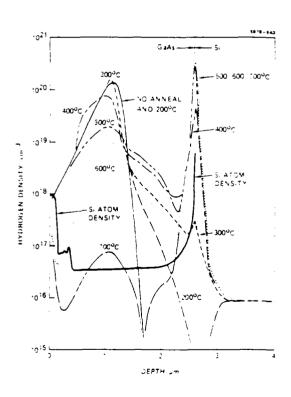


Fig. 24. Redistribution of H in a GaAs/Si structure

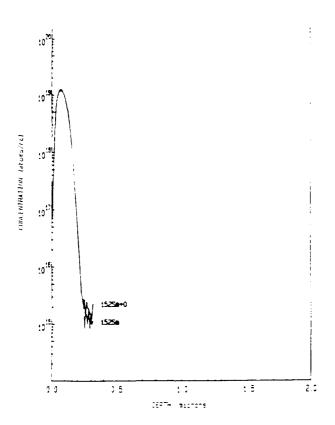


Fig. 25. Random depth profile of Sm implanted into GaAs

Fig. 26. Channeled depth profile of Sm implanted into GaAs

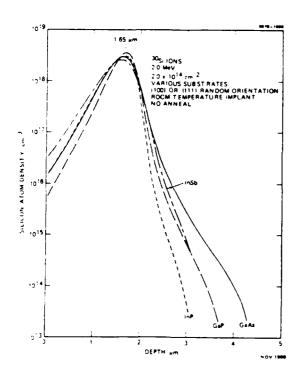


Fig. 27. Depth profiles of MeV Si in GaAs, GaP, InP, and InSb

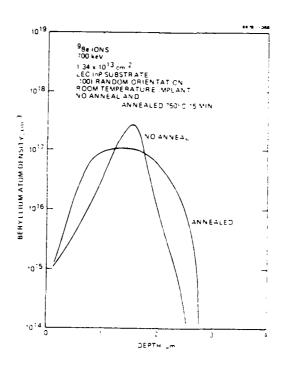


Fig. 28. Depth profiles of 0.7MeV Be in InP

APPENDIX

On following pages are summary listings of work performed that is related to this contract effort. Included are lists of implants and anneal conditions, by implanted element and by material, a list of anneal temperatures by sample number, and a list of art code numbers by sample number. Finally, brief summaries of measurements made and results obtained are given by sample number, for some, but not all samples.

Summary of Hydrogen [¹H and ²H (deuterium)], He, Be, Si, and other Implants into Various Substrates (primarily III-V materials and $LiNb0_3$) made for and during the ARO Contract. "A" means that annealing studies (temperature series) were performed (See next table for temperatures.).

ID No.	Mat'l Dopant/S	L Source	Energy keV	Fluence	Orient ID	Anneal
H3118 H3119 H3391 H3477 H3478 H3479 H3519 H3534 H3535 H3590	GaAs Si GaAs Si GaP S GaP S InP S InP Sn GaAs Zn GaAs SI GaAs SI LiNbO3		200 200 300 333 333 333 300 50 50	5.0E15 5.0E15 5.0E15 5.0E15 5.0E15 1.0E16 5.0E15 1.0E16	<110> <100> 111 111 -11 100 -A 100 -A 711 712A	A A A A A A
D103 D104 D105 D204 D205 D206 D207 D208 D209 D210 D211 D212 D213 D214 D215	GaAs GaAs GaAs GaP GaP InP InP InP InP InP InP InP InP InP In	HRL	200 100 50 100 200 350 100 200 350 350 200 100 100 200 350	3.0E15 3.0E15 3.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15 2.0E15	110 110 110 100 100 100 110 110 111 111	A A
D216 D217 D218 D219 D226 D227 D228 D229 D230	GaAs Si GaP S InP Sn InP S GaSb - GaAs Zn GaAs SL He+GaAs GaAs/Si Si	HRL HRL HRL HRL Pearton Pearton	350 350 350 350 300 300 300 75 100	5.0E15 5.0E15 5.0E15 5.0E15 5.0E15 1.0E16 1.0E15 5.0E15	110 111 -F A 100 -D A 100 -E A A 20 He 123 MOCVD 10236	A A A A
D231 D232 D239 D240 D241 D245 D247 D281 D282	InSb AlAs/GaAs SL InSb/GaAs SL InSb GaAs none LEC InSb/GaAs SL GaAsP LiNbO ₃ LiNbO ₃	Comas Pearton HRL	300 300 300 300 300 200 150 100	5.0E15 5.0E15 5.0E15 5.0E15 5.0E15 5.0E15 5.0E15 1.0E16	LN ₂ 971 LN ₂ (see D231 1000s	A A A A

D283 D284 D285 D286	GaP GaP InP InAs	Zn S S	Pearton HRL HRL Pearton	300 300 300 300	8.5E15 8.5E15 8.5E15 8.5E15			A A A
He123 He126 He131 He136 He137 He138	GaAs GaAs GaP GaP InP GaP	? S	HRL HRL HRL HRL HRL	300 200 100 200 350 350	3.0E15 3.0E15 3.0E15 3.0E15 5.0E15 5.0E15	100 100 111 111 100 111	-B -D	A A
Be2579 Be2580 Be2608 Be2666 Be3312 Be3316 Be3317 Be3318 Be3319 Be3322 Be3323	InP GaP,InP GaAs ARO GaAsP InP GaSb LiNbO ₃ GaAs GaAs GaP InSb	SL SL Fe	HRL HRL Comas Novak Novak HRL Pearton Xtal Technol Airt Pearton	700 700 700	5.0E15 5.0E12 1.0E14 2.0E14 1.3E13 2.0E13 1.3E13 1.3E13 1.3E13 1.3E13 1.3E13	100	-B 712B	A A A A A A A
Be3348 C228-240	InAs O(part)	various	Pearton HR!	200 200 400 700	1.OF14 2.OE13 2.OE13 2.OE13 2.OE13			
0886-93	2(part)	various	HRL	100 200 400 700	2.0E13 2.0E13 2.0E13 2.0E13			
Mg289-29	92(part)	various	HRL	250	1.0E14			
Si9171 Si9500 Si9667 Si9667	GaSb GaAsP InAs InP		Pearton Novak Pearton 28 HRL +30	150 300 180 180	2.0E13 1.0E14 1.0E14 2.0E14			A A
S976-102	27(part)	various	HRL	100 200 400 700	2.0E13 2.0E13 2.0E13 2.0E13			
Zn157-17	71(part)	various	HRL	200 400	2.0E13 2.0E13			

Se577-588(part) various	HRL	200 200 400 400	4.0E12 1.0E14 4.0E12 1.0E14	
Se589-602(part) various	HRL	200	2.0E13	
Se634-640(part) various	HRL	400	1.OE14	
Cd298-301(part) various	HRL	500	1.0E14	
Y2-3 GaAs,InP	HRL	300	1.0E14	
La2-3 GaAs, InP	HRL	300	1.0E14	
Nd2-3 GaAs, InP	HRL	300	1.0E14	
Sm2-3 GaAs,InP	HRL	300	1.0E14	
Er2-3 GaAs,InP	HRL	300	1.0E14	
Yb2-3 GaAs,InP	HRL	300	1.0E14	
MeV Implants MeV H LiNbO ₃	Xtal Technol	1000	3.0E15	A
GaAs(Si)	ARO	1000	3.0E15	A A
GaP(S)	HRL	1000	3.0E15	Ā
InP(S)	HRL	1000	3.0E15	A
InP(Fe)	HRL	1000	3.0E15	A
InSb	ATT	1000	3.0E15	A
MeV He GaAs	••••	2000	5.0E15	A
GaP	n	2000	5.0E15	Ā
InP		2000	5.0E15	A
$LiNbO_3$		2000	5.0E15	A
MeV Si GaAs		2000	2.0E14	A
GaP	n	2000	2.0E14	A
InP		2000	2.0E14	A
InSb		2000	2.0E14	
LiNbO ₃		2000	2.0E14	A
MeV Ge GaAs	_	2400	1.0E15	A
GaP	n	2400	1.0E15	A
InP		2400	1.0E15	A
InSb		2400	1.0E15	

```
783, 847R1, -932, -933, 934, 848, 849, -841, -787,
Be2534-56
                                        846R1, 935, 845, 930
Be2608
              - 909
Be2579
               844R1
Be2580
               845, 846R1
Be3316 InP
              -1088
Be3317 GaSb
              -1091
Be3318 \ LiNb0_3 -1099
Be3319 GaAs
              -1089
Be3322 GaP
              -1090
Be3323 InSb
              -1091
Si9500
              -1092
Se
             -928(GaP)
MeV Si(6)
             -942, -1098
MeV Ge(2.4)
             -949(GaP), -950, -951
```

Summary of Implants by Material

GaAs(p) GaAs(n) GaAs	H3519 H3118, H3119, MeV H D229, D241, He123, Be3 Y2-3, La2-3, Nd2-3, Sm	319	Yb2-3
GaP(p) GaP(n)	H3391, H3477, MeV H He138	D283 D217, D284	Be2579,80 Be3322
InP(p) InP(n)	H3478, H3479, MeV H He139	D208, D209,	D218, D219, D285
InP(- or Fe)	He137, Be2579, Be2580 Y2-3, La2-		9667 2-3, Er2-3, Yb2-3
InAs	MeV H	D286	Be3348 Si9667
GaSb		D226	Be3317 Si9171
InSb	MeV H	D231, D240	Be3323
GaAs/Si	D230		
GaAsP		D247	Be3312 Si9500
AlGaAs SLs	H3534	D228, D232	Be2608, Be2666
InSb/GaAs SLs	H3535, MeV H	D239, D245	
LiNbO ₃	H3590, MeV H	D281, D282	Be3318

Summary of Annealing Temperatures

Temperatures (in °C) done as part of annealing studies performed for the samples listed in the Implant Summary table.

```
100, 200, 300, 400, 500, 600
H3391
H3477
        300, 325, 350, 375, 400, 450, 500
H3478
        300, 325, 350, 375, 400, 450, 500, 550, 600
H3479
        300, 325, 350, 375, 400, 450, 500, 550, 600
H3519
        100, 200, 300, 350, 400, 450, 500, 600
H3535
        300, 500
        250, 300, 350, 400
H3590
H 1.0 MeV set 300, 500
D216
        350, 400, 450, 500
D217
        300, 325, 350, 375, 400, 450, 500, 550, 600
                                                        +350, 500 FL
D218
        275, 300, 325, 350, 375, 400, 450, 500, 550, 600
                                                        +350, 500 FL
D219
        275, 300, 325, 350, 375, 400, 450, 500, 550, 600
        200, 225, 250, 275, 300, 400, 500, 600
D226
D227
        200, 250, 300, 350, 400, 500, 600, 700
D228
        200, 300, 400, 500, 600, 700
D229
        (He123) 300, 500
D230
        200, 300, 400, 500, 600
        100, 150, 175, 200, 225, 250, 275, 300, 400, 500
D231
D239
        150, 200, 250, 300
D240
        100, 125, 150, 175, 200, 300, 400, 500
D241
        200, 250, 300, 350, 400, 450, 500, 550, 600, 700
D281
        250, 300, 350, 400
D282
        300, 500, 700
        300, 350, 400, 500, 600, 700
D283
D284
        300, 500, 700
D285
        300, 500, 700
        300, 500, 700
D286
```

He 2.0 MeV set 300

Be profiles in annealed SL 712B Be2608 300, 500, 550, 600 Be set at 700 keV 750

Si MeV set 750

Summary of Implant/Annealing/SIMS Profiling Studies by Implant/Sample Number

H3118 GaAs 200 keV 5.0×10^{15} cm⁻² <110> CH Annealed 300, 500°C H3119 GaAs 200 keV 5.0x10¹⁵ cm⁻² <100> CH Annealed 300, 500°C This experiment was performed to determine how channeled implants or channeled depth distributions redistribute upon annealing whether they redistribute differently from random implant profiles. Both show good redistributed profiles for 300°C anneal - both deeper and toward the surface; the <100> profiles are like the random profiles, but the <110> shallow, toward the surface profile is somewhat different, as if there were less effect of damage, which there should be because the implant was channeled. The deep diffused profile is displaced deeper for the <110> implant, essentially by the deeper channeled beginning location of the H depth distribution for the diffusion. For 500°C, the H profiles are just detectable, that is, nearly gone in both crystal orientations, but visible after background subtraction in the region of the peak of the original profile.

- H3535 ARO Superlattice 712 (Comas) implanted with 5.0×10^{15} cm⁻² 50 keV ¹H. Annealed at 300 and 500°C.
- H3477 GaP(S) Implanted with 5x10¹⁵ cm⁻² 333 keV ¹H. Annealed at 350, 375, 400, 450, 500°C. The deep H diffusion profiles are just great enough in density to detect, using background subtraction (10¹⁷-10¹⁸ cm⁻³). The shallow redistribution toward the surface begins at 375°C and is quite high in density (>10¹⁹ cm⁻³) even to 500°C. Thus GaP retains H in this region to annealing temperature of greater than 500°C.

H3478 InP(S) Implanted with 5x10¹⁵ cm⁻² 333 keV ¹H. Annealed at 325, H3479 InP (Sn) 350, 375, 400, 450, 500, 550, 600°C. Both n-type. Both same H profiles. The deep H diffusion profile in both is too low in density to detect(<10¹⁷ cm⁻³). The shallow redistribution toward the surface is easily detected - H density between 10¹⁹ and 10¹⁷ cm⁻³. H reaches the surface at 375°C, and then decreases to <10¹⁷ cm⁻³ by 600°C.

H3519 GaAs(Zn) from Pearton implanted with 1.0x10¹⁶ cm⁻² 333 keV ¹H.

Annealed at 100, 200, 300, 350, 400, 450, 500, and 600°C. Shows the redistribution of H toward the surface and deeper into the bulk, because the Zn-doped p-type GaAs retains H at a much higher density -- above the detection limit of SIMS for ¹H. The H does not disappear from the GaAs until 600°C is reached.

H3535 Comas AlGaAs/GaAs superlattice 712, implanted with 5x10¹⁵ cm⁻² 50-keV ¹H and annealed at 300 and 500°C. Shows the Al layer oscillations, the H profiles, and H decoration of the strain or dislocations at the superlattice/substrate interface. H is nearly gone in the SL at 500°C, but remains at the buffer layer/substrate interface.

D208 840 Comb of 838 and 839

D214 840

D213 100 keV InP from HRL implanted with 2.0x10¹⁵ cm⁻² ²H.

D214 200 keV An energy set.

D215 350 keV Profile prameters shown below for 200 and 350 keV.

	200 keV	350 keV
$R_m \mu_m$	1.96	3.34
$R_p \mu m$	1.90	3.28
$\Delta R_p \mu m$	0.216	0.296
γ_1	-1.26	-0.83
β_2	6.91	5.68
$oldsymbol{eta_2}$,	0.91	0.68
$N_{\text{max}} \text{cm}^{-3}$	4.6x10 ¹⁹	4.8x10 ¹⁹

- D226 GaSb matrial from S. Pearton of AT&T Bell Labs, implanted with 5x10¹⁵ cm⁻² 300 keV ²H and annealed at 100, 200, 225, 250, 275, 300, 350, /JO, and 500°C. H redistribution, beginning at 200°C, is shown to progress deeper into the substrate with increasing annealing :emperature; the H is gone for the 400 and 500°C temperatures.
- D227 GaAs(Zn) material from S. Pearton of AT&T Bell Labs, implanted with 5×10^{15} cm⁻² 300-keV ²H and annealed at temperatures of 100, 200, 300, 400, 500, 600, and 700°C. The H redistribution is generally the same, except in the region of heaviest implant damage, under the peak of the implant, where several peaks or an oscillation in the H profile is seen.
- D229 LEC GaAs from HRL implanted with 5×10^{15} cm⁻² 300-keV He, and subsequently implanted with 1×10^{15} cm⁻² 75-keV ²H and annealed at 300 and 500°C. No redistribution occurs for 300°C, but for 500°C, the H decorates the damage from the He implant, with some structure.
- D230 MOC'D epi GaAs on Si, doped with Si, from Spire Corp. via S. Pearton of AT&T, implanted with 5×10^{15} cm⁻² 100-keV ²H, and annealed at 200, 300, 400, 500, 600, and 700°C. The Si profile through the GaAs layer was measured. The H redistributes beginning above 200°C, and decorates the strain or dislocations at the GaAs/Si interface. H is still present in the GaAs layer, but at reduced density, after 700°C.
- D231 InSb from S. Pearton of AT&T Bell Labs, ~50°C implanted with 5x10¹⁵ cm⁻² 300-keV ²H, and annealed at 200, 250, 300, and 350°C. All temps show redistribution, even the as-implanted sample. H is gone (beneath detection limit) by 350°C. See D240.
- D240 Same as D231, except implanted at -150°C, and annealed at 125, 150, and 175°C. H redistributes more like in GaAs toward surface and deeper with increasing temperature.
- D241 GaAs(Si) from ARO. Implanted with 5x10¹⁵ cm⁻² 300 keV ²H. Annealed at 200, 300, and 400°C.

- D245 InSb/GaAs MBE superlattice from J. Comas of NRL/NBS (972,973: 10 and 20 nm layers of 1.5μm total thickness), implanted with 5x10¹⁵ cm⁻² 200-keV ²H, and not yet annealed. We have not been successful in measuring the alternate layers of this difficult structure using low energy (1.5 keV) 0 SIMS, so disordering cannot be studied.
- D281 LiNbO₃ from HRL implanted with 5.0×10^{15} cm⁻² 100 keV ²H and annealed at 300, 350, 400, and 450°C. These profiles show the striking redistribution of H in LiNbO₃ that include the "uphill" diffusion and the high density of H in diffused LiNbO₃ >10¹⁸ cm⁻³.
- D282 LiNbO $_3$ from Univ. of Glasgow implanted with 1.0x10 16 cm $^{-2}$ 100 keV 2 H. This implant served as the SIMS standard for quantification of the H SIMS profiles in the Univ. of Glasgow hydrogen exchanged and annealed LiNbO $_3$ work.
- D283 GaP(Zn) from S. Pearton of ATT Bell Labs. Implanted with 8.5x10¹⁵ cm⁻² 300 keV ²H. Annealed at 300, 350, 400, 500, 600, and 700°C. Shows H redistribution well because it is Zn-doped p-type GaP, which retains a high density of H. More anneal temperatures are being done now and the set will be completed after this report is submitted. These results should be published.
- D284 GaP(S) from HRL. Implanted with 8.5x10¹⁵ cm⁻² 300 keV ²H. Annealed at 300, 500, and 700°C. Shows H redistribution but at lower density than D283.
- D285 InP(S) from HRL. Implanted with 8.5x10¹⁵ cm⁻² ²H. Annealed at 300, 500, and 700°C. MORE. Also, S was profiled under the ²H implant for the 300 and 500°C anneals. S shows oscillations of ~1μm period that may be S decoration of strain induced in the InP crystal during LEC growth as the temperature controller cycles and the temperature varies periodically very slightly.
- D286 InAs from S. Pearton of ATT Bell Labs. Implanted with 8.5x10¹⁵ cm⁻² 300 keV ²H. Annealed at 300, 500, and 700°C MORE